MichiganEngineering

In-Situ SEM Investigation of Microstructural Damage Evolution and Strain Relaxation in a Melt-Infiltrated SiC/SiC Composite

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40th Annual Conference on Composites, Materials, and Structures

Motivation – Experimental Data for Modeling

- Robust CMC life prediction capabilities require experimental data for inputs and validation
- Several groups working on environmental degradation models that incorporate matrix cracking and interface debonding
  - few studies report measured crack opening displacements
- NASA GRC characterizing CMCs to support environmental modeling

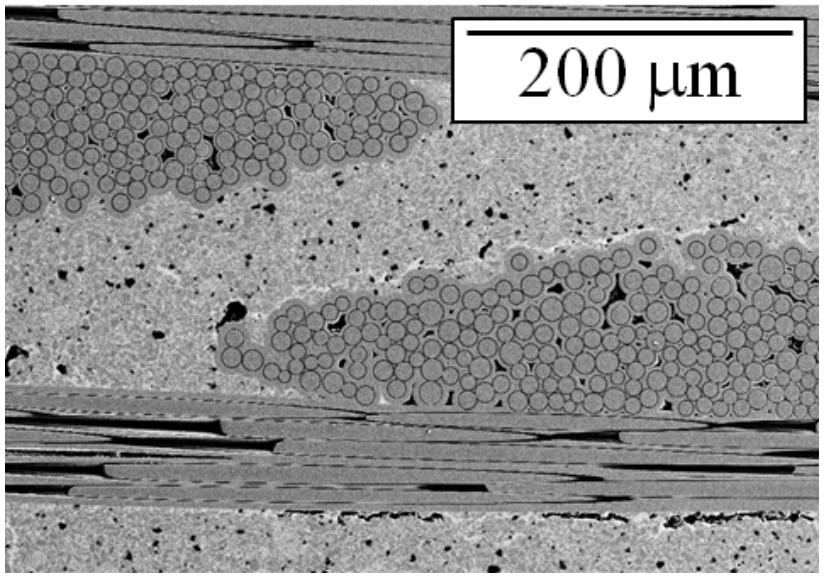
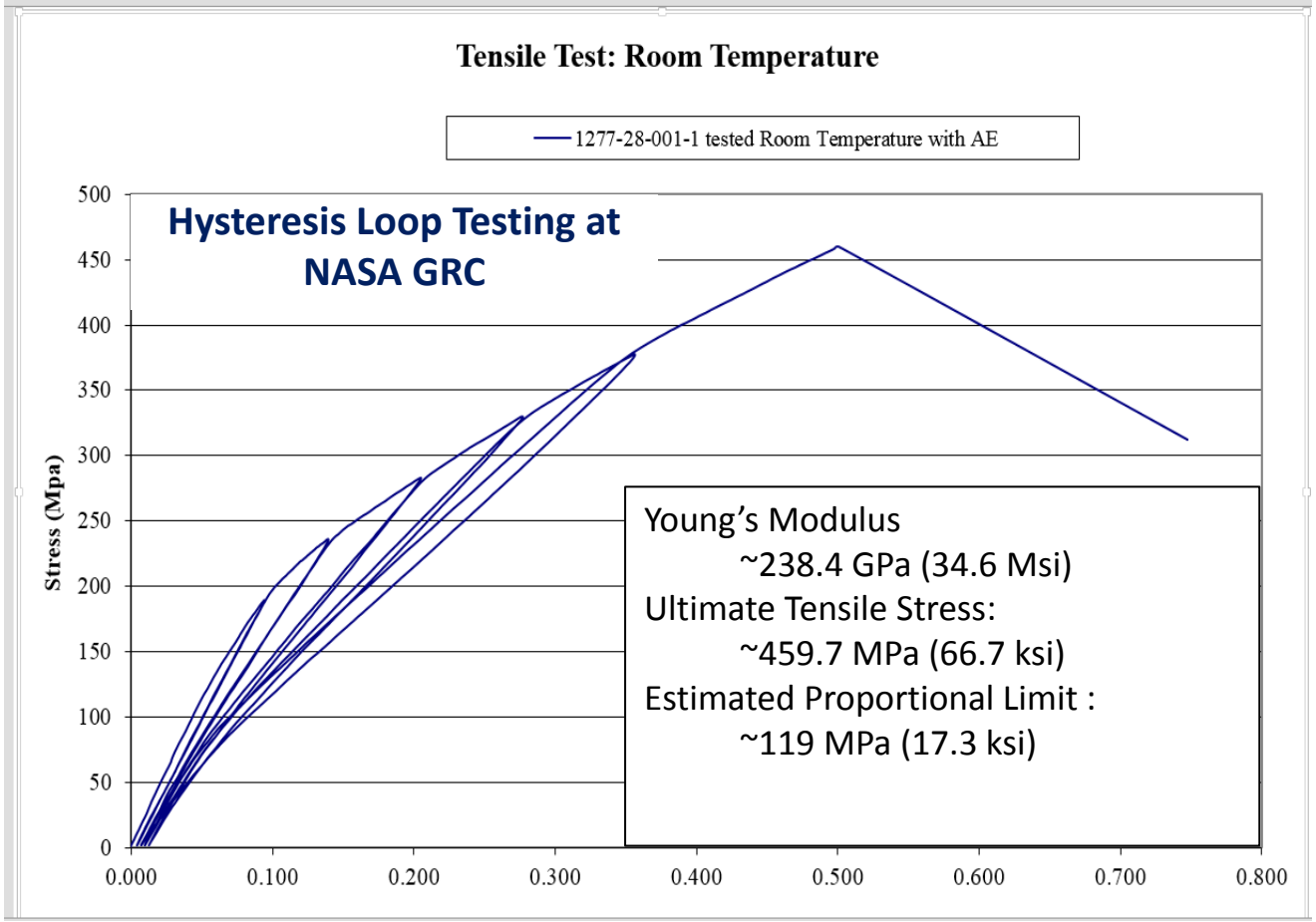


Image Courtesy of NASA GRC

  - Sylramic fiber reinforced, slurry cast MI SiC/SiC
  - CODs predicted to be very - small – too small for traditional DIC
  - Apply SEM-DIC using small tensile loading stage in SEM

Approach



- Load in 5 ksi increments to 30 ksi using small tensile stage
- Measure COD using SEM-DIC and manual methods

Digital Image Correlation

Non-contact "optical" method

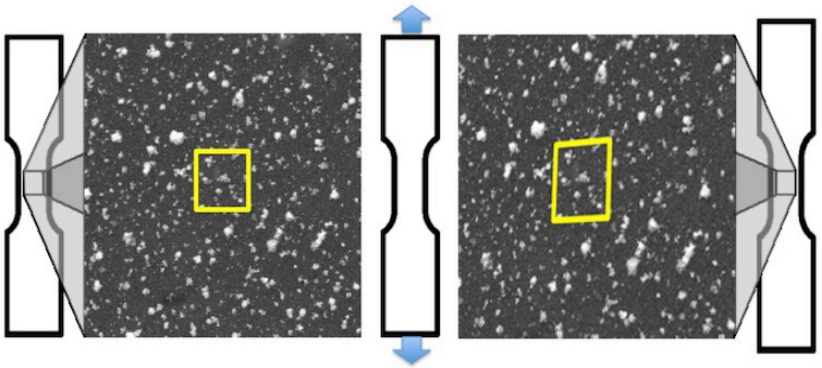
White light, SEM, AFM

Requires surface to have a random tracking pattern

Isotropic, high-contrast, random

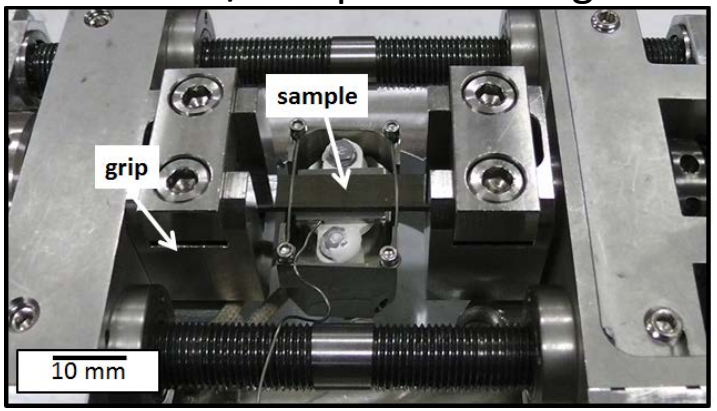
Surface pattern analyzed in small subsets

Grayscale intensity within subsets is tracked as sample is deformed.

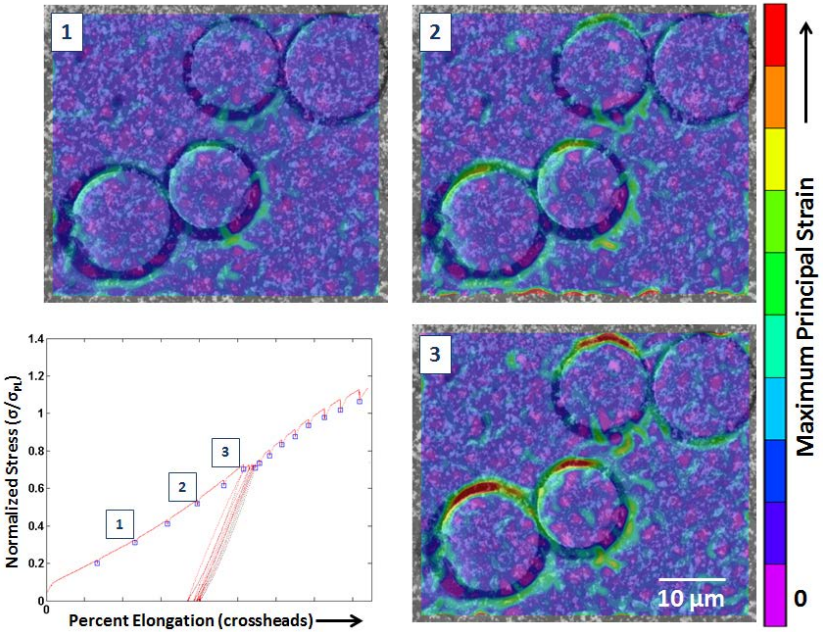


Sutton M.A., et al. (1983), *Image and Vision Computing*, **1(3)**: 133-139.
Bruck, H.A., et al. (1989), *Exp. Mech.*, **29**: 261-267.

In-SEM Miniature Tension/Compression Stage

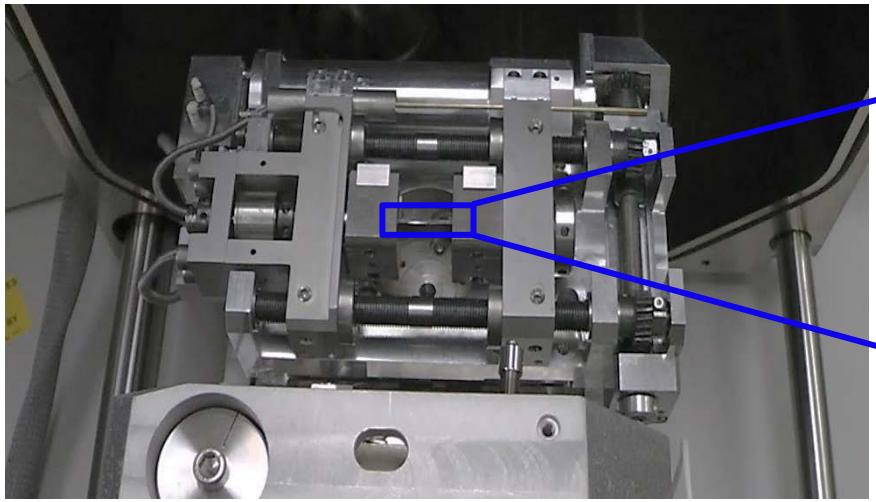


SEM-DIC applied to CMCs

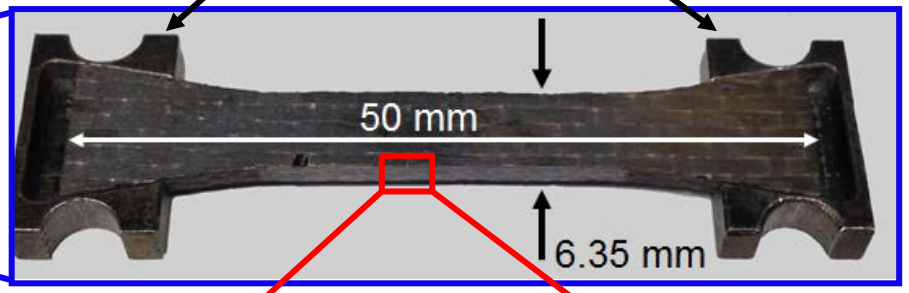


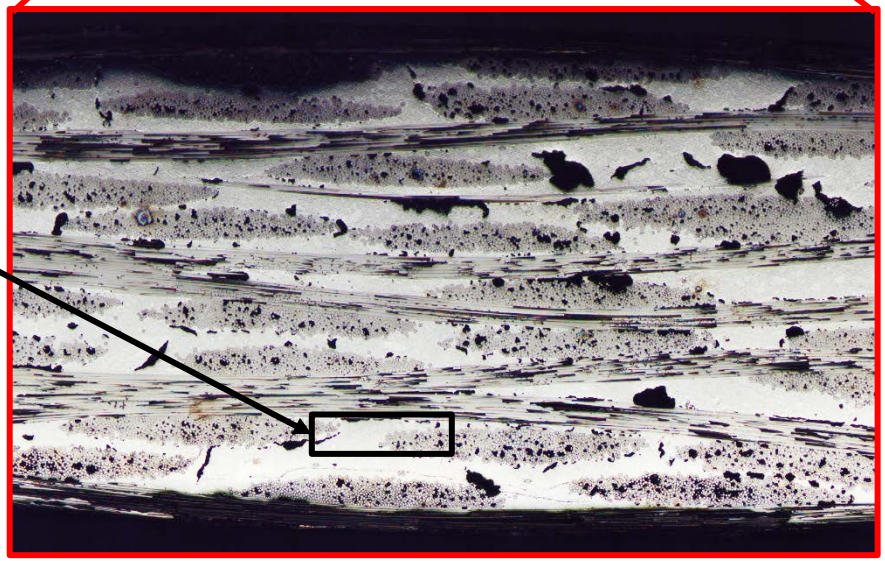
Tracy, J.M., et al. Cocoa Beach 2014

Loading and Imaging Configuration



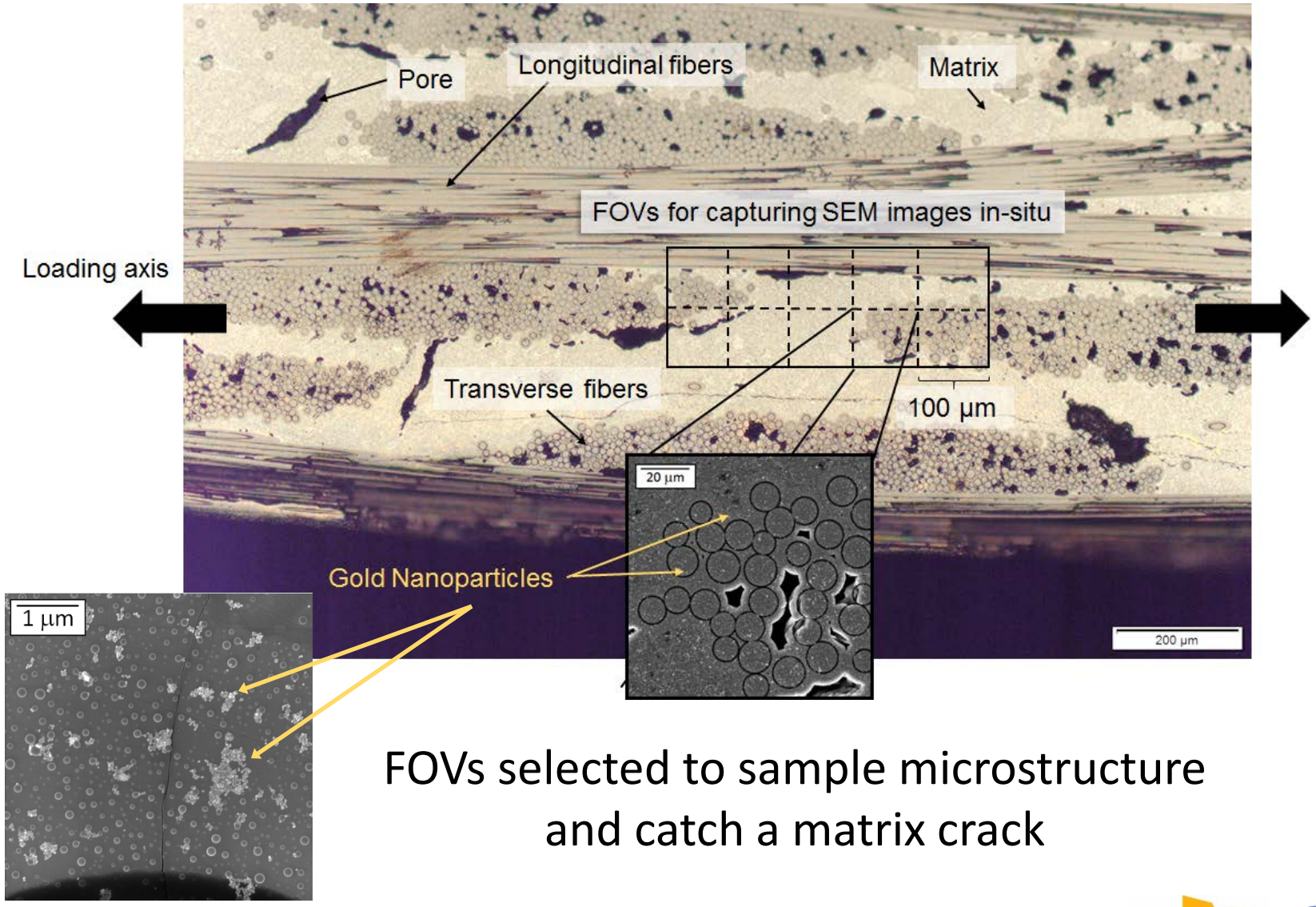
Loading Direction





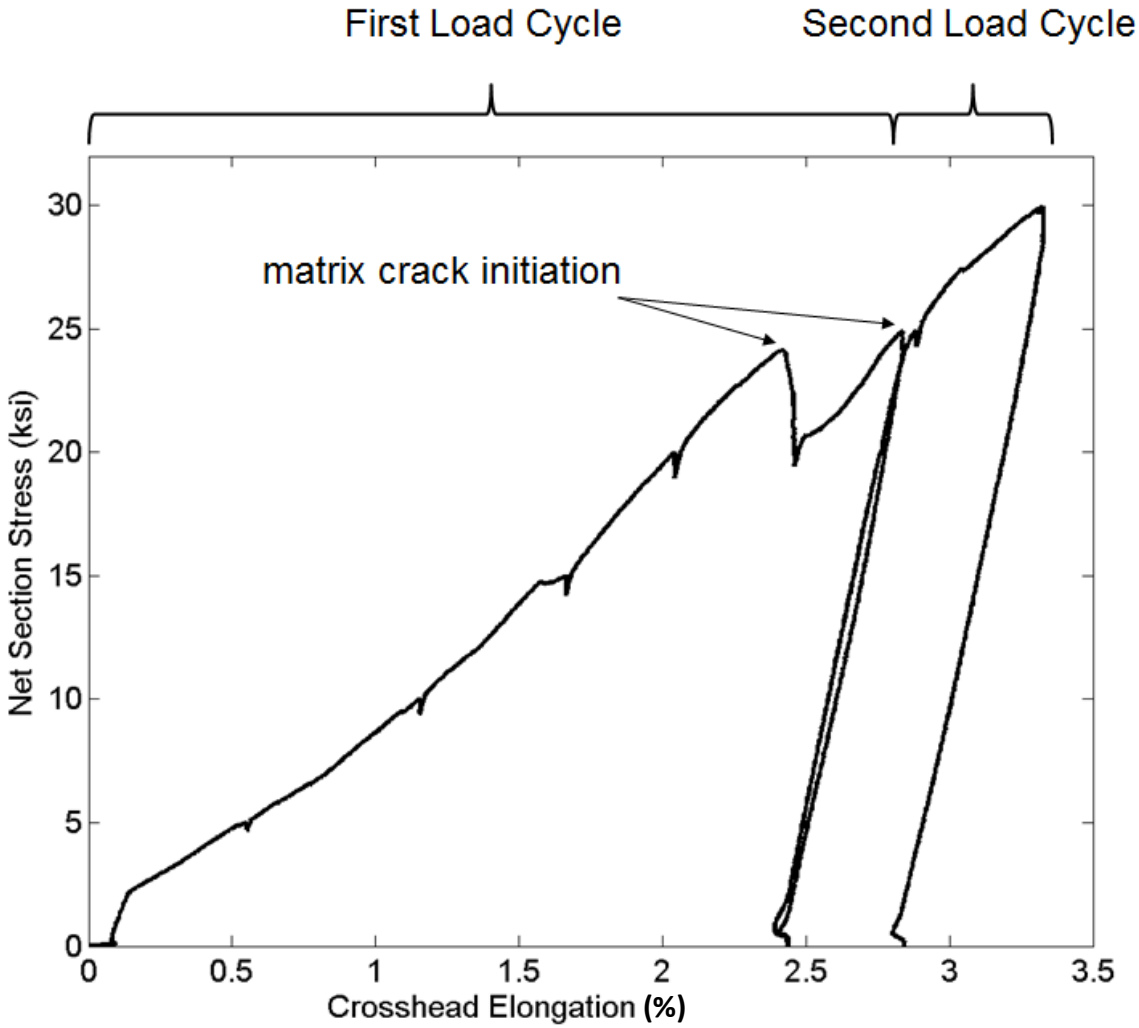
Area analyzed was a 200 µm x 500 µm rectangle located ≈ 1.4 mm left and 0.7 mm below centroid of gage section

Initial Fields of View (FOVs)

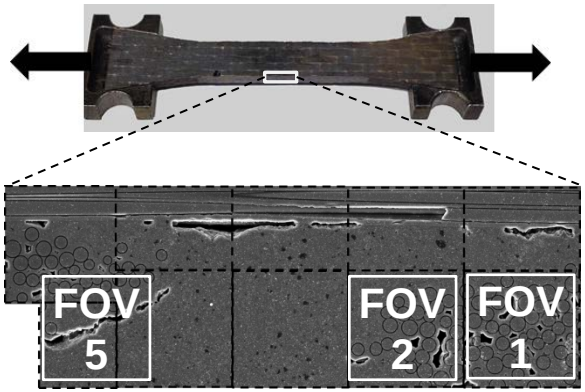


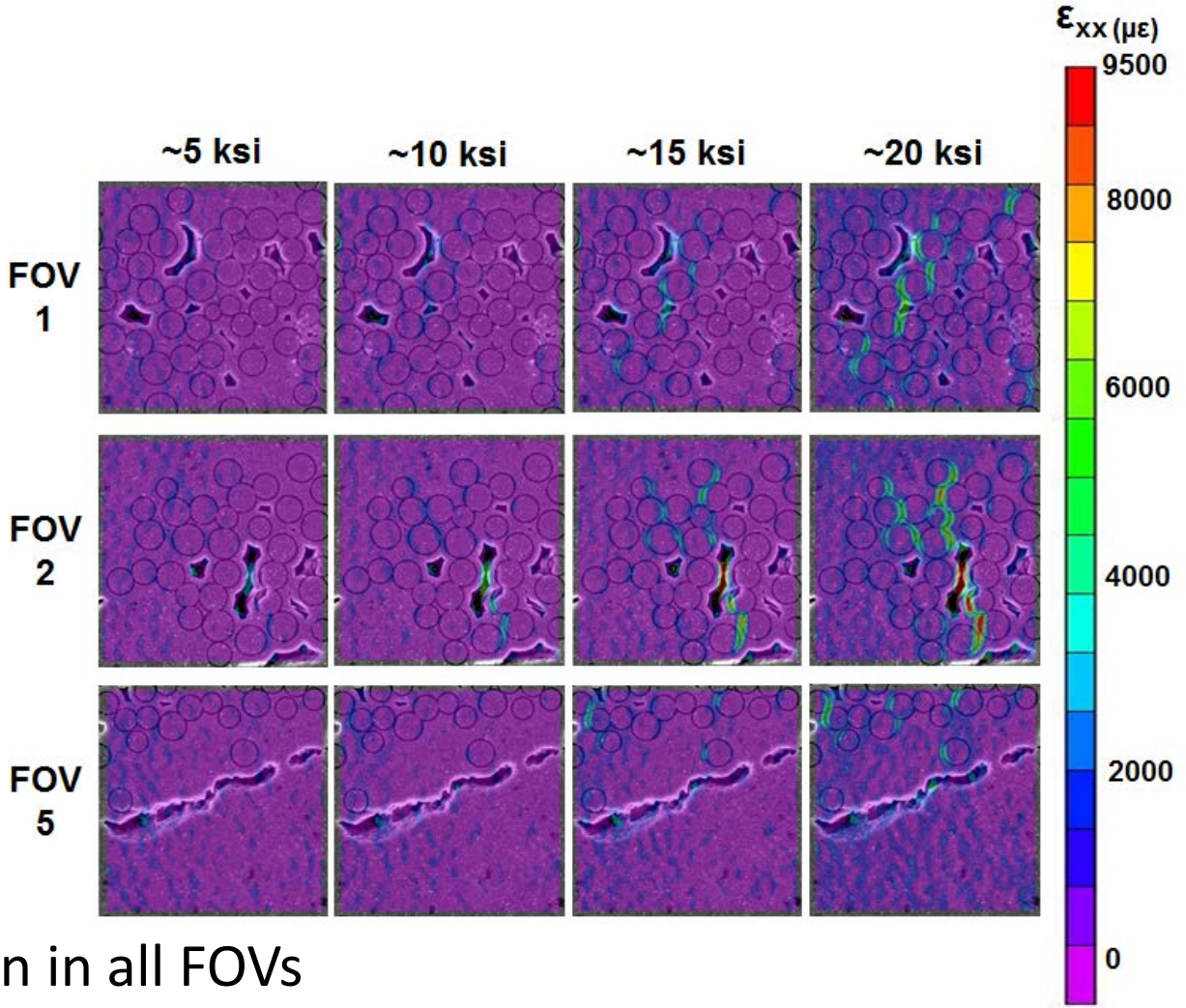
FOVs selected to sample microstructure and catch a matrix crack

- Sample loaded in tension at ~5 ksi stress increments
- Loading paused at each stress increment to capture SEM images
- Images captured after load relaxed
- Matrix cracks formed between 20 and 25 ksi of initial load cycle, but outside of imaging area.
- Sample unloaded/reloaded to capture matrix crack openings displacements in new AOI



Damage Evolution Before Matrix Cracking



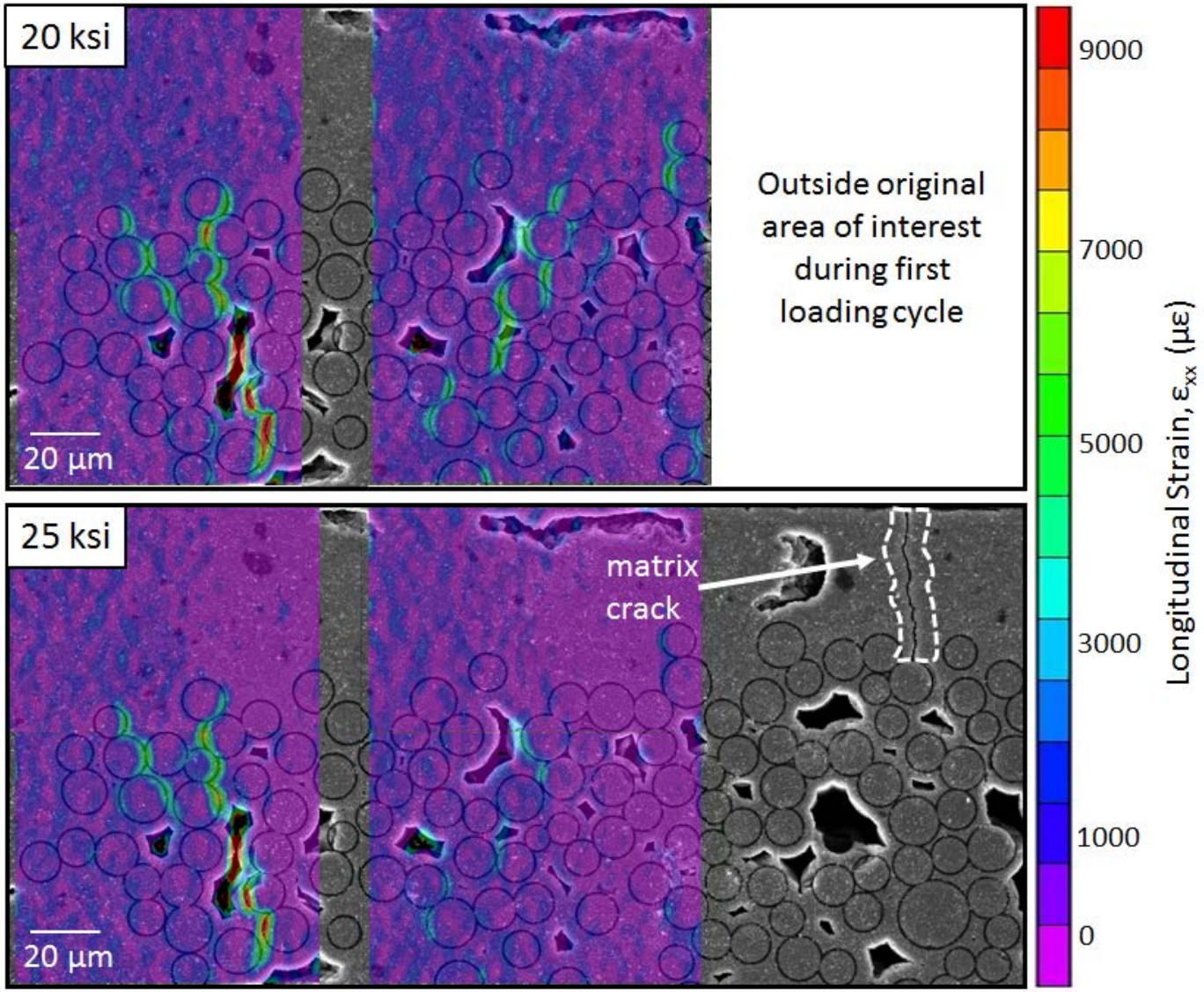


- Strain localization seen in all FOVs
- Strain localization observed ~ 10 ksi

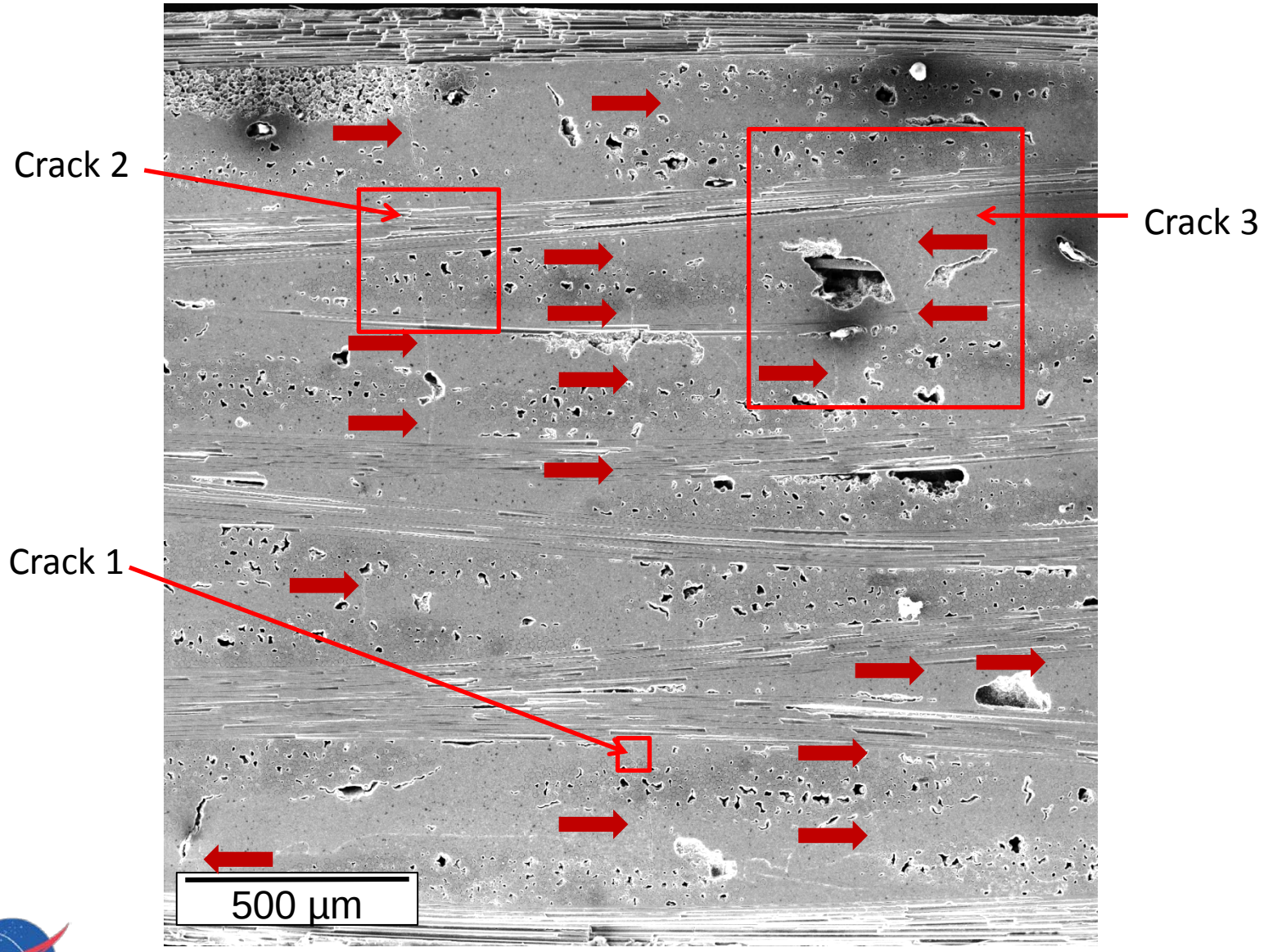
Strain Relaxation Adjacent to Matrix Crack

Prior to first matrix cracking

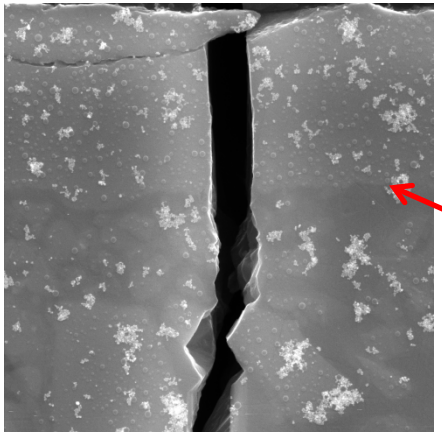
After first matrix cracking



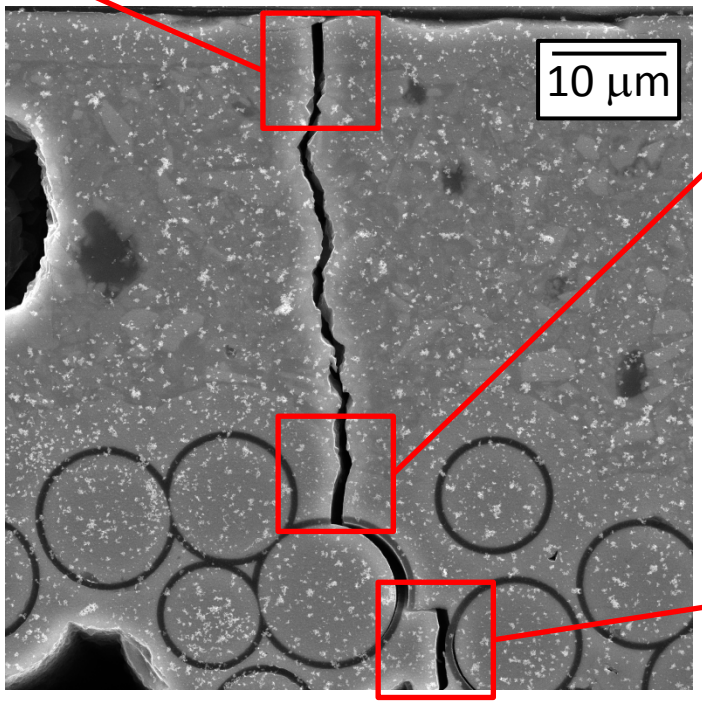
Cracks observed across the cross-section

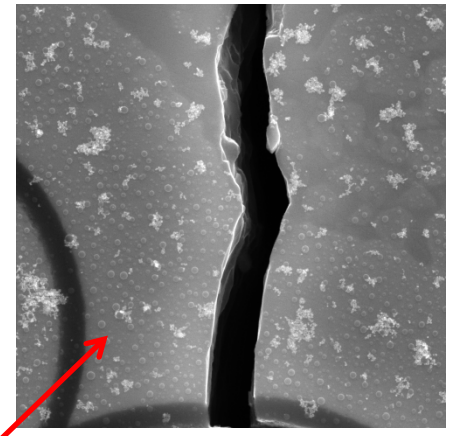


Crack 1

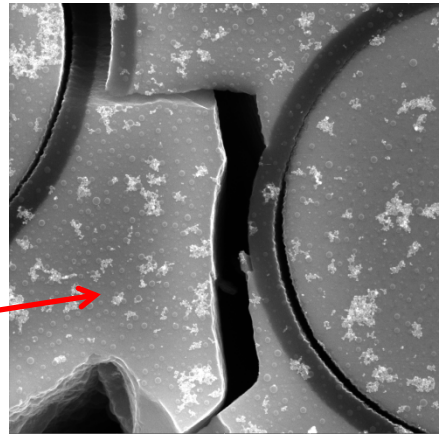


Location 2



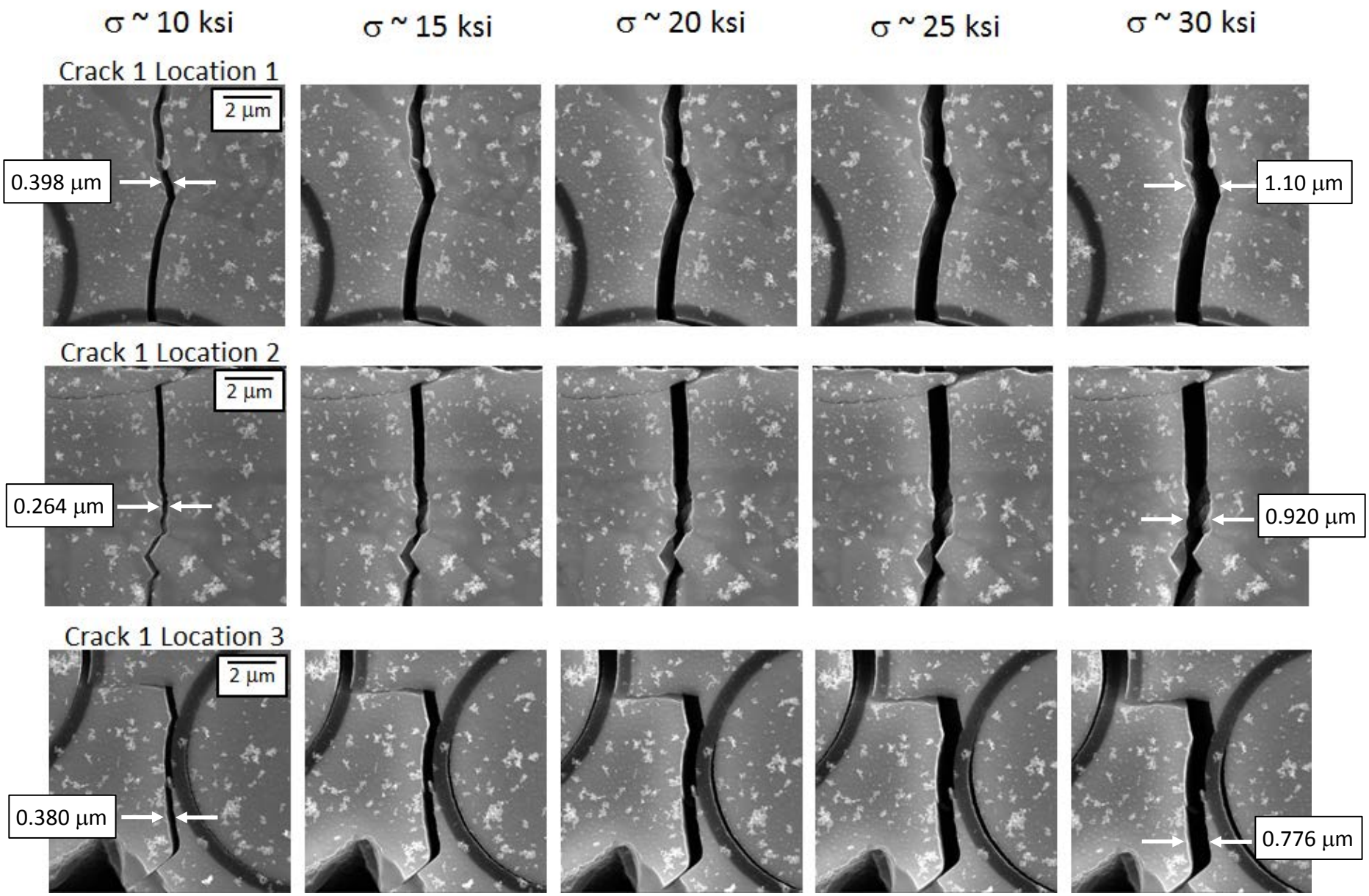


Location 1

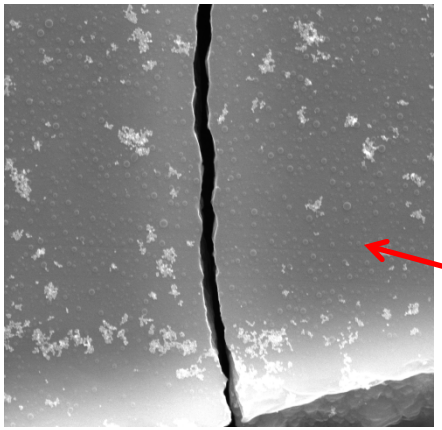


Location 3

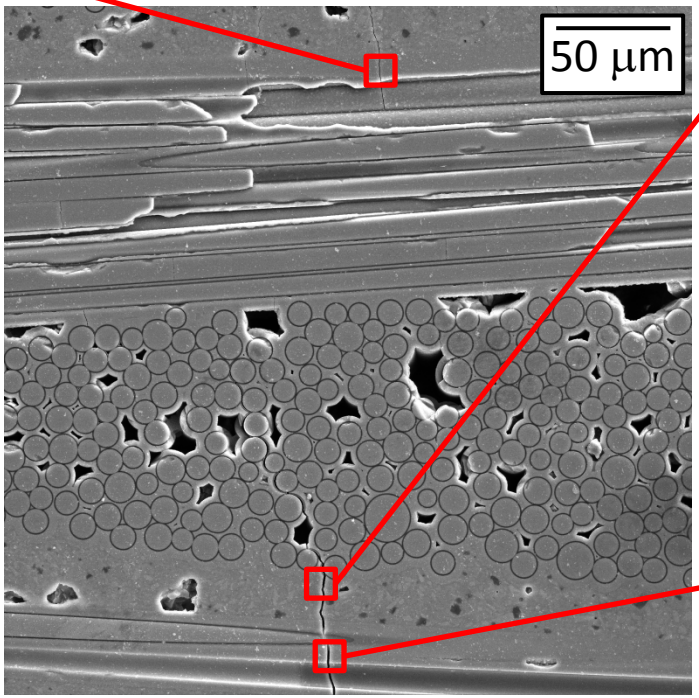
- All high mag FOVs are 10 µm
- High mag FOVs shown at ~30ksi

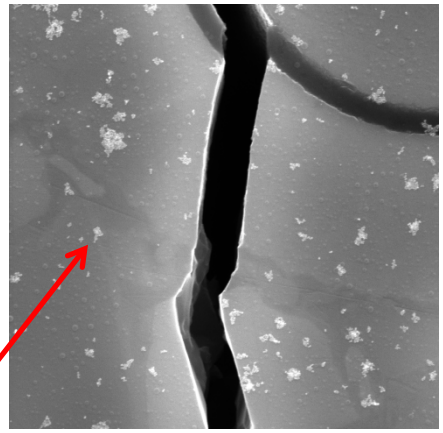

σ ~ 10 ksi
σ ~ 15 ksi
σ ~ 20 ksi
σ ~ 25 ksi
σ ~ 30 ksi
Crack 1 Location 1
2 µm
0.398 µm
1.10 µm
Crack 1 Location 2
2 µm
0.264 µm
0.920 µm
Crack 1 Location 3
2 µm
0.380 µm
0.776 µm

Crack 2

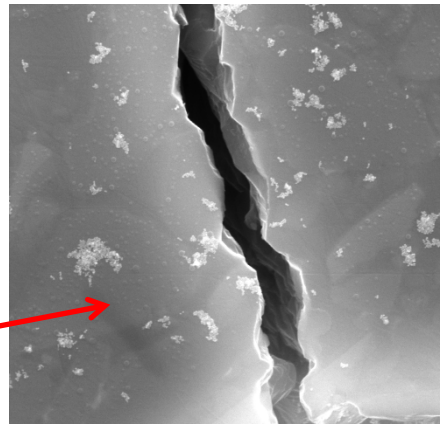


Location 3



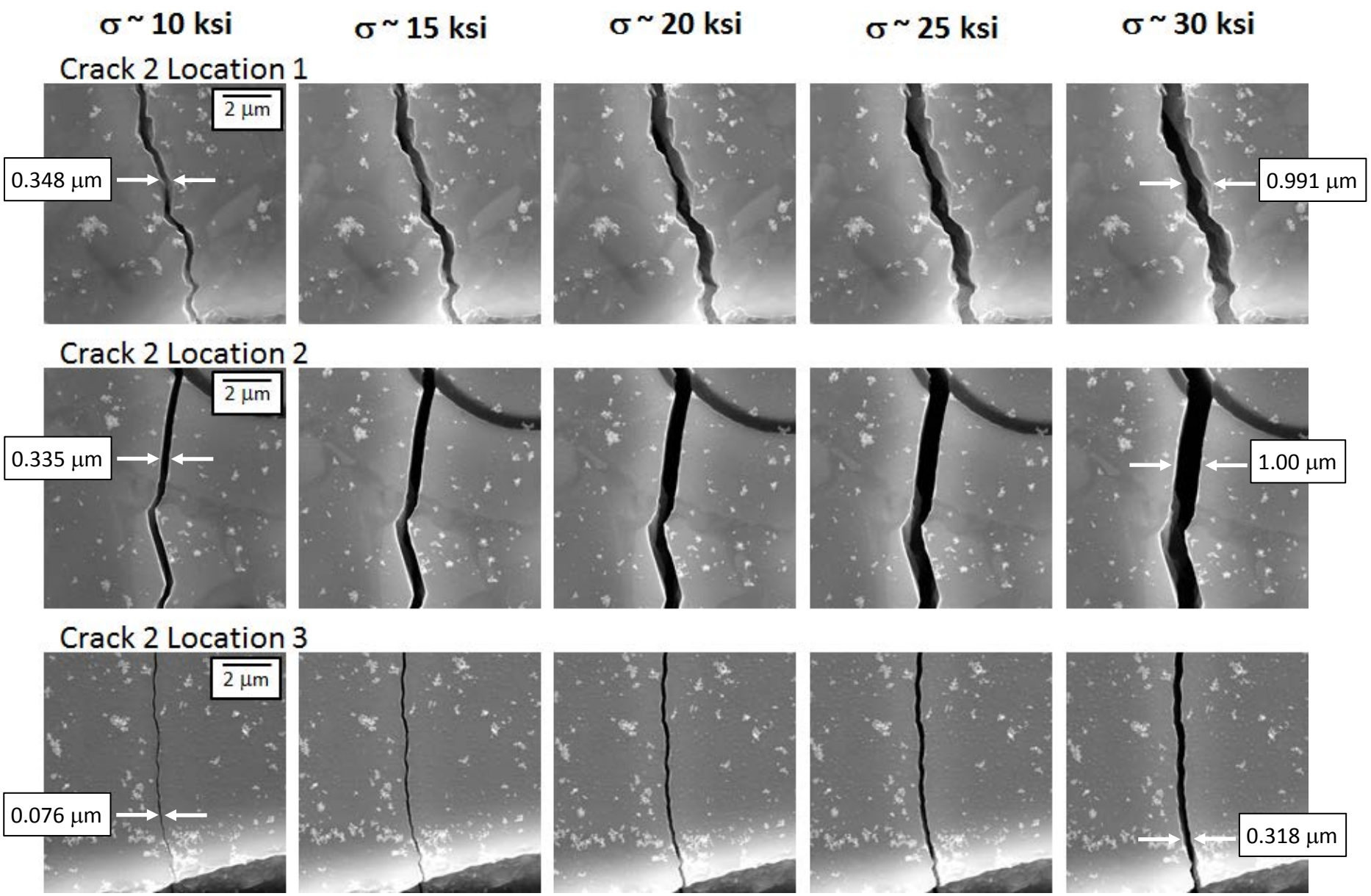


Location 2

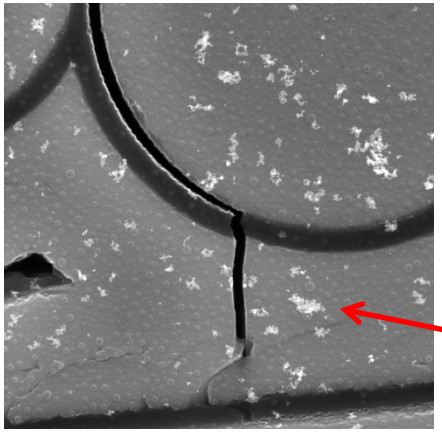


Location 1

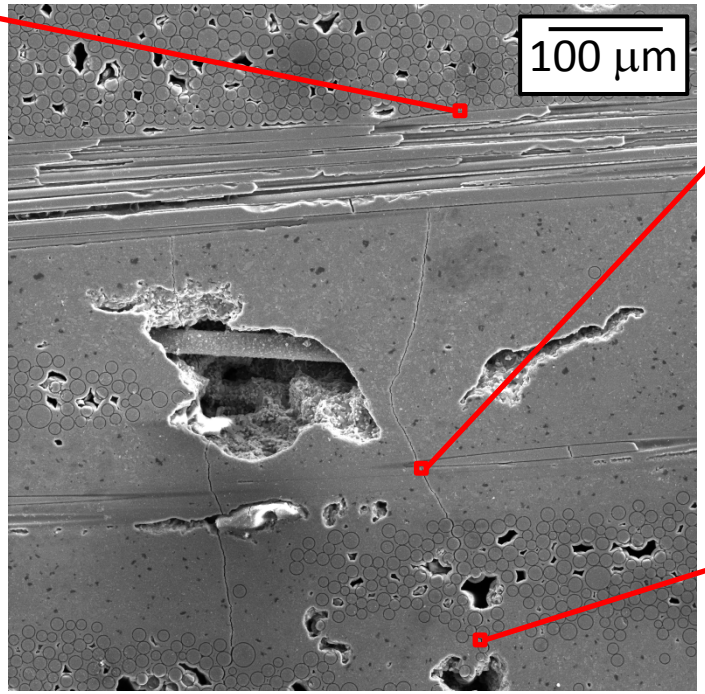
- All high mag FOVs are 10 µm
- High mag FOVs shown at ~30ksi

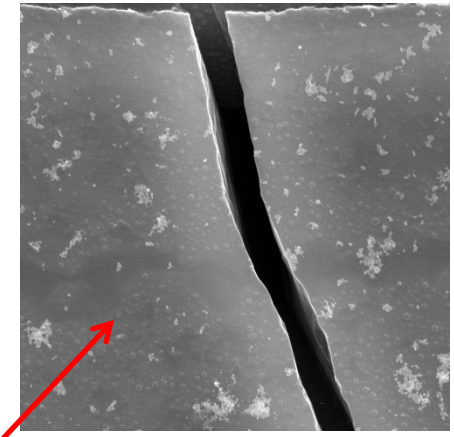

σ ~ 10 ksi
σ ~ 15 ksi
σ ~ 20 ksi
σ ~ 25 ksi
σ ~ 30 ksi
Crack 2 Location 1
2 µm
0.348 µm
0.991 µm
Crack 2 Location 2
2 µm
0.335 µm
1.00 µm
Crack 2 Location 3
2 µm
0.076 µm
0.318 µm

Crack 3

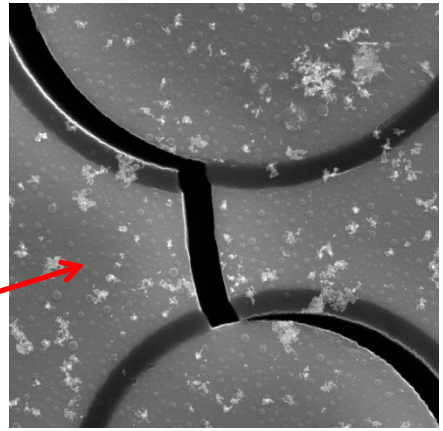


Location 3



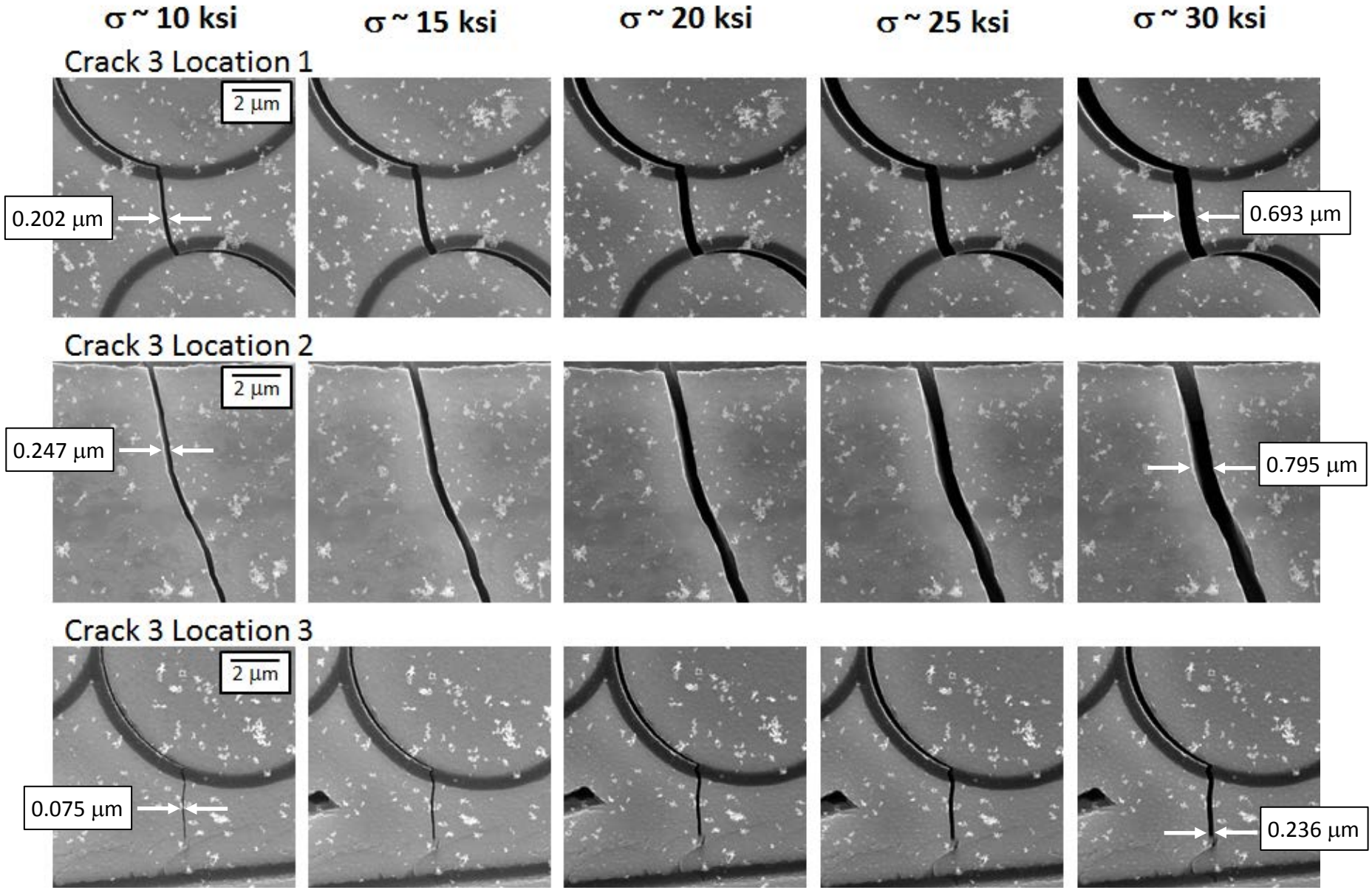


Location 2

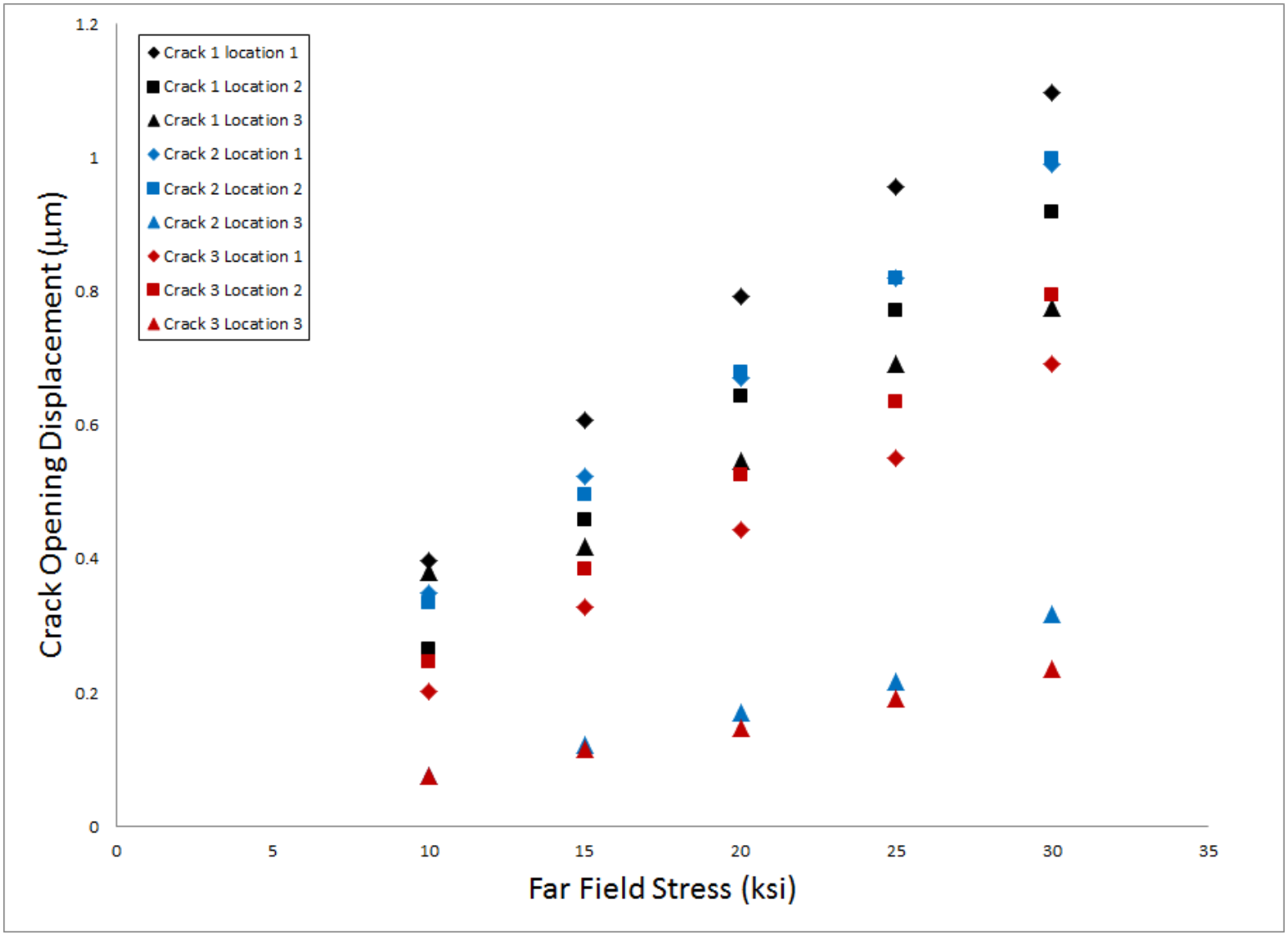


Location 1

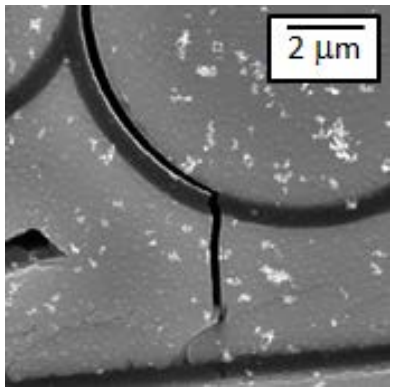
- All high mag FOVs are 10 µm
- High mag FOVs shown at ~30ksi

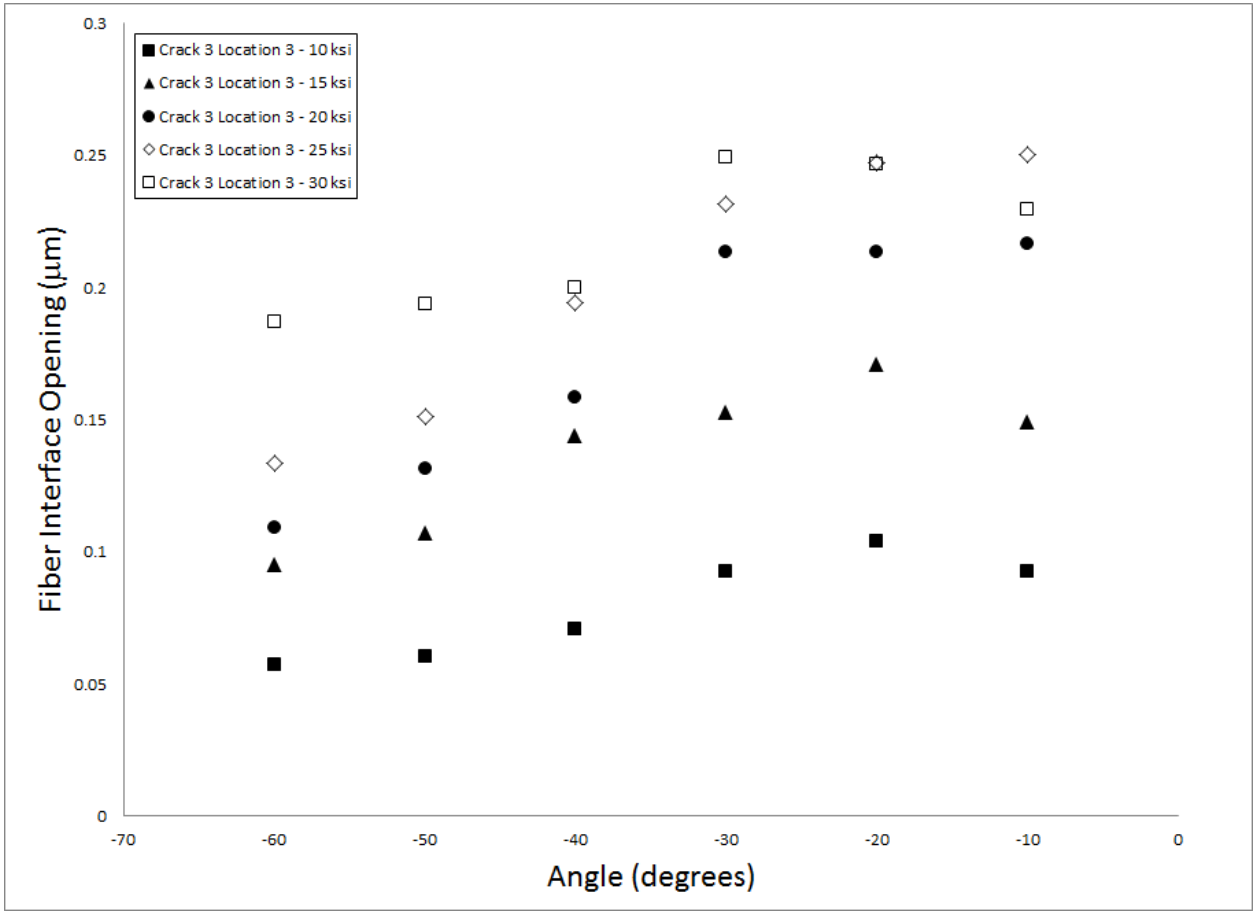

σ ~ 10 ksi
σ ~ 15 ksi
σ ~ 20 ksi
σ ~ 25 ksi
σ ~ 30 ksi
Crack 3 Location 1
2 μm
0.202 μm
0.693 μm
Crack 3 Location 2
2 μm
0.247 μm
0.795 μm
Crack 3 Location 3
2 μm
0.075 μm
0.236 μm

Matrix Crack Opening Exhibits Variability



Interface Opening Exhibits Variability

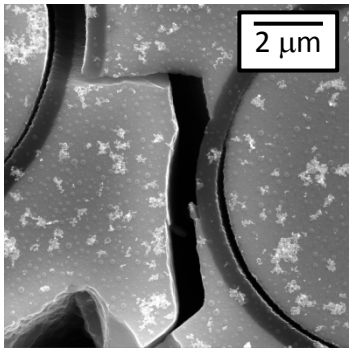




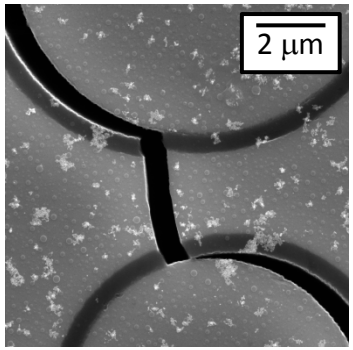
- Expect opening max along direction of stress max
- Stress component along opening direction =$\sigma\cos(\theta)$

Cracking Along Interfaces

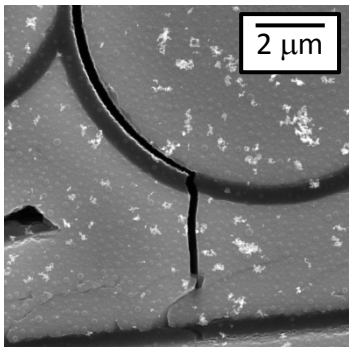
Crack 1 Location 3

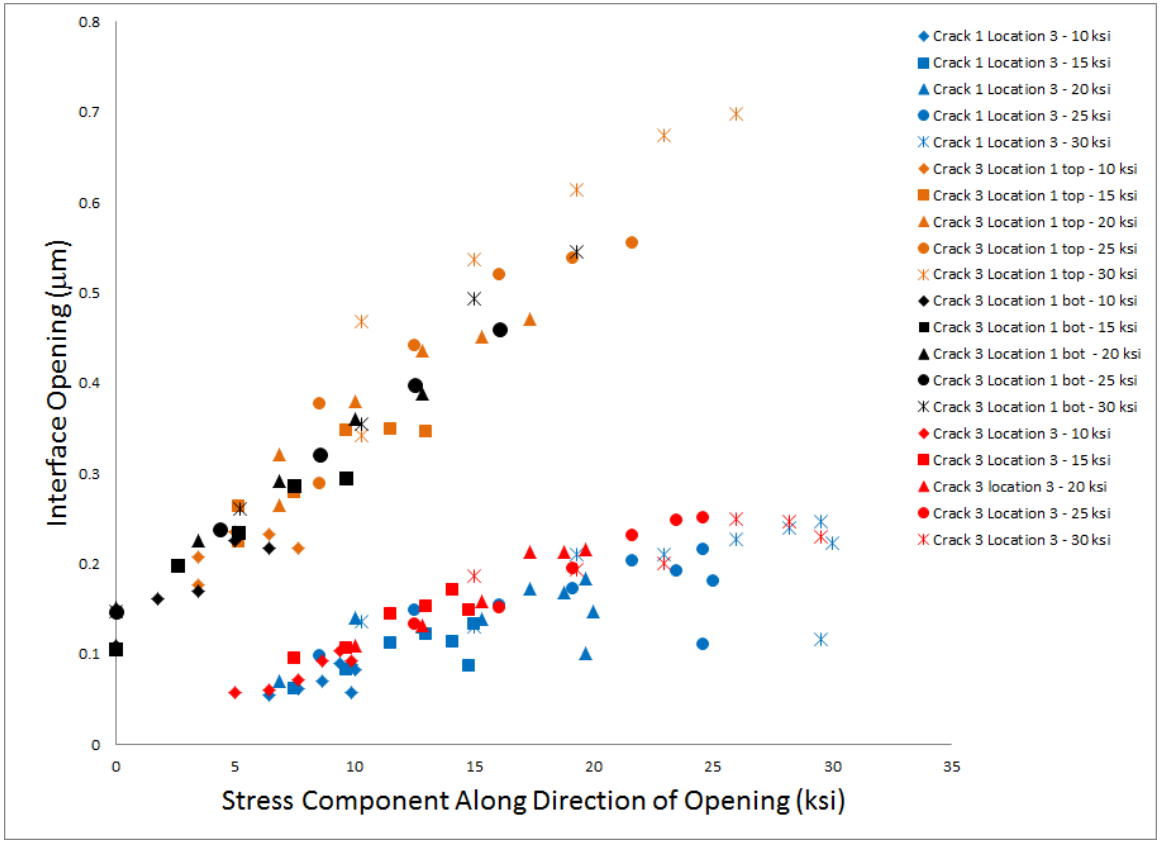


Crack 3 Location 1



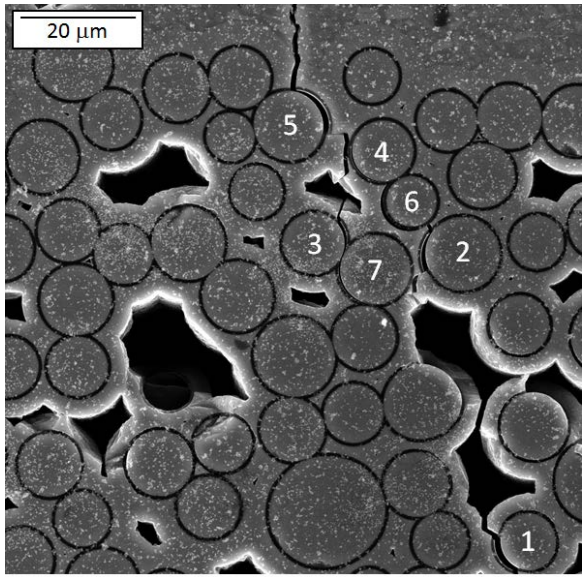
Crack 3 Location 3

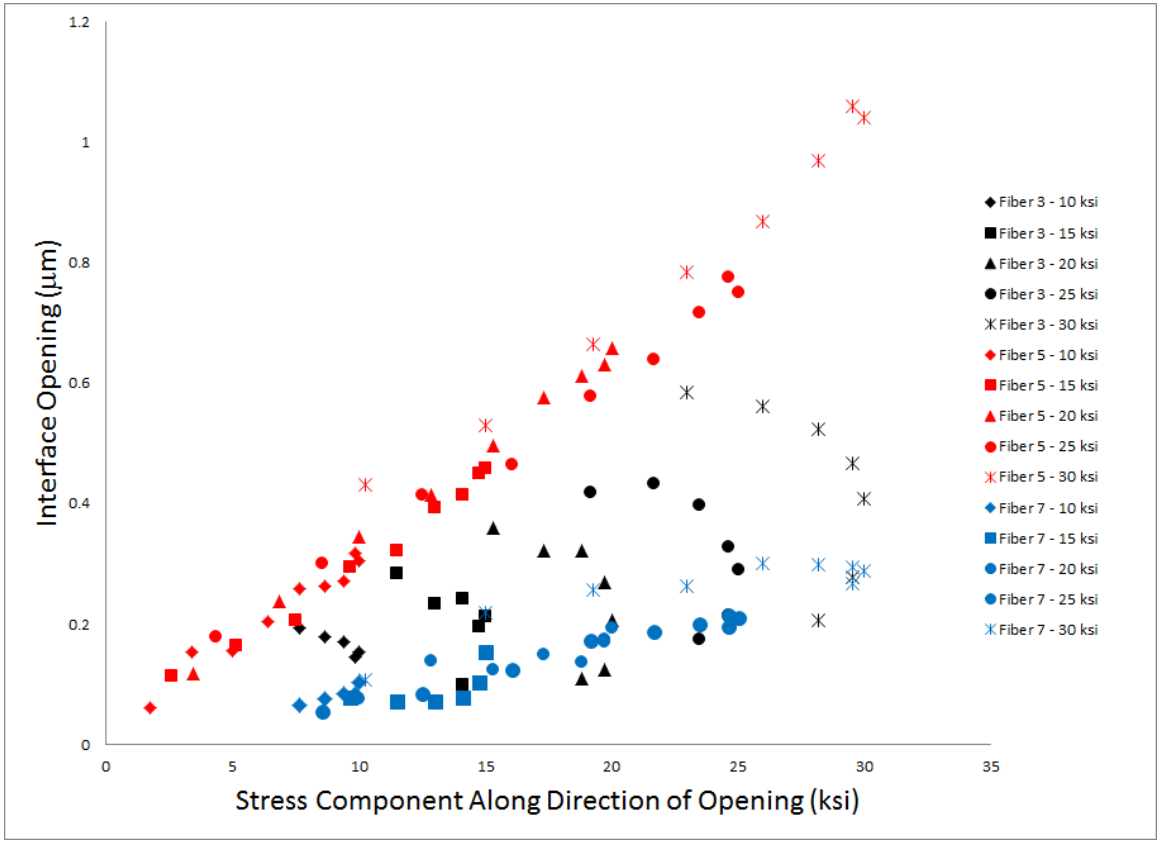




- Some openings follow max global stress, some do not
- Cannot see the entire opening in the FOV

Multiple Fibers Along Crack 1





- Again some openings follow max global stress, some do not
- Local stress state is unknown

Future Work

In-situ Testing and Analysis

- Couple macroscale DIC with SEM-DIC to examine the multiscale nature of damage evolution and the influence of microstructure on crack growth
  - Couple high speed imaging with macroscale DIC to examine and quantify the distances over which matrix cracks influence neighboring cracks
- SEM-DIC at ultrafine length scales (FOVs < 5 μm) to probe mechanical response in matrix constituents – available constituent properties are mostly approximations
- Examine environmental effects on subcritical crack growth
  - investigate the effects of fatigue, humidity, combustion gases on crack growth in both coatings and matrix
  - SEM/ESEM (microscale) or an environmental chamber (macroscale)

Modeling

- Statistical modeling of the influence/impact of microstructural features on damage evolution (for data collected in all of the above studies)
  - Quantify and correlate measurements of microstructural features with damage observations
  - Use results to develop models describing the influence of microstructure on damage evolution.

Summary and Conclusions

- A slurry cast MI SiC/SiC sample was loaded to a global stress of 30 ksi in a small tensile stage within an SEM
- SEM-DIC and traditional analysis was used to quantified damage
- Damage at fiber/matrix interfaces at global stresses as low as 5 ksi
- After initial matrix cracking, strain relaxation was observed adjacent to matrix cracks
- Crack opening displacements varied from 0.2 to 1.5 μm at a global stress of 30 ksi
- Interface openings exhibited angular variability where maximum opening was not always along the global loading axis - opening may follow a local maximum